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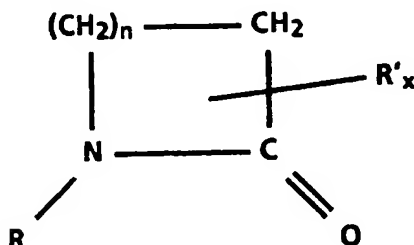
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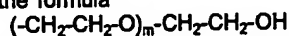
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(54) Ink jet compositions and processes.

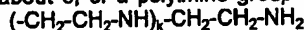
(57) Disclosed is a thermal ink jet printing process which comprises incorporating into a thermal ink jet printing apparatus an ink composition comprising a colorant and a liquid vehicle which comprises a mixture of water and an organic component selected from the group consisting of : (1) cyclic amides of the formula



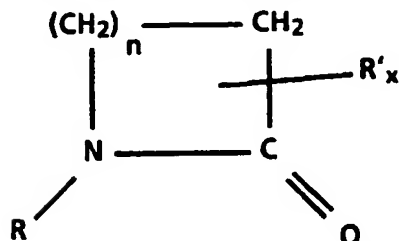
wherein n is a number from 2 to about 12 ; R is hydrogen, alkyl or substituted alkyl with from 1 to about 6 carbon atoms, a polyethoxy group of the formula



with m being a number of from 0 to about 9, or a polyimine group of the formula

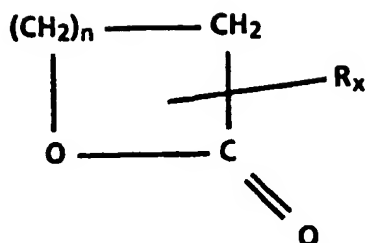


with k being a number of from 0 to about 9 ; R' represents one or more substituents that can replace hydrogen in any of the hydrocarbon portions of the molecule, and is an alkyl group, a halogen atom, a sulfate group, a nitro group, a sulfone group, an amide group, or an acetyl group, wherein x is a number of from 0 to $2n + 2$; (2) a cyclic amide of the formula

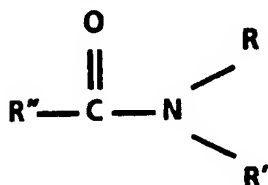


wherein n is a number of from 1 to about 12, R is a cyclohexyl group or a butyl group, and R' represents one or more substituents that can replace hydrogen in any of the hydrocarbon portions of the molecule, and is an alkyl group, a halogen atom, a sulfate group, a nitro group, a sulfone group, an amide group, or an acetyl group, wherein x is a number of from 0 to $2n + 2$; (3) cyclic esters of the formula

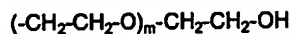
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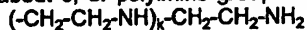
wherein n is a number of from 1 to about 12, R represents one or more substituents that can replace hydrogen in any of the hydrocarbon portions of the molecule, and is an alkyl group, a halogen atom, a sulfate group, a nitro group, a sulfone group, an amide group, or an acetyl group, wherein x is a number of from 0 to $2n + 2$; (4) amides of the formula



wherein R and R' are alkyl groups or substituted alkyl groups with from 2 to about 20 carbon atoms, polyethoxy groups of the formula



with m being a number of from 0 to about 9, or polyimine groups of the formula



with k being a number of from 0 to about 9, wherein R and R' can be bonded to each other to form a ring, wherein R can also be hydrogen and wherein R' is hydrogen or alkyl, with alkyl preferably having from 1 to about 20 carbon atoms; and mixtures thereof; and heating selected nozzles in the printing apparatus containing the ink, thereby causing droplets of the ink to be ejected in an imagewise pattern onto a substrate.

The present invention relates to ink jet printing processes and ink compositions suitable for said processes.

Known ink jet inks generally comprise a water soluble dye which is soluble in an ink vehicle such as water or a mixture comprising water and a water soluble or water miscible organic solvent. For example, US-A-4,849,770 (Koike et al.) discloses an ink for use in an ink jet comprising a reactive dye or a reactive dispersing dye and a solvent composed mainly of water and an organic solvent which is not reactive with the dye. US-A-4,793,264 (Lin et al.) discloses an ink for use with an ink jet system having material subject to corrosion by the ink, said ink comprising a fatty acid vehicle, a colorant, and amounts of anti-oxidant. Further, Japanese Patent Publication 1-158083 discloses an ink composition for ink jet recording comprising 0.1 to 10 weight percent of a colorant, which can be either a dye or a pigment, 1 to 10 weight percent of a penetrating agent, and 25 to 98.9 weight percent of a polar solvent, such as formamide, DMSO, dimethyl ethanolamine, or N-methyl-2-pyrrolidone. In addition, US-A-3,994,736 (Hertz et al.) discloses a pigment-free high intensity light fast ink suitable for ink jet printing which comprises a polar solvent liquid base and a premetallized azo dye. The ink may also contain an inorganic ionizable salt to impart electrical conductivity.

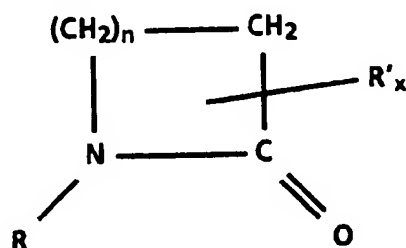
Heterophase ink jet inks are also known. For example, US-A-4,705,567 (Hair et al.) discloses a heterophase ink jet ink composition which comprises water and a dye covalently attached to a component selected from the group consisting of poly(ethylene glycols) and poly(ethylene imines), which component is complexed with a heteropolyanion. In addition, US-A-4,597,794 (Ohta et al.) discloses an ink jet recording process which comprises forming droplets of an ink and recording on an image receiving material by using the droplets, wherein the ink is prepared by dispersing fine particles of a pigment into an aqueous dispersion medium containing a polymer having both a hydrophilic and a hydrophobic construction portion.

Other relevant prior art on ink compositions, some of which are especially suitable for use in ink jet printers, includes US-A-4,395,287, 4,196,006, 4,170,482, 4,073,615, 4,990, 186, 5,006,172, 4,620,876, JP-A-51/008031.

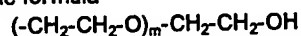
While known compositions and processes are suitable for their intended purposes, a need remains for ink compositions suitable for use in ink jet printers. In addition, a need remains for ink compositions with rapid drying times. A need also remains for ink compositions that exhibit long latency times in ink jet printers. Further, there is a need for ink compositions that produce sharp, non-feathering printed characters on plain paper. There is also a need for ink compositions that enable high print quality while also providing rapid drying times and/or long latency times in ink jet printers.

It is an object of the present invention to provide improved ink compositions and processes which meet these needs.

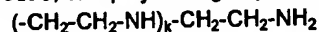
These and other objects of the present invention can be achieved by providing a thermal ink jet printing process which comprises incorporating into a thermal ink jet printing apparatus an ink composition comprising a colorant and a liquid vehicle which comprises a mixture of water and an organic component selected from the group consisting of: (1) cyclic amides of the formula



wherein n is a number of from 2 to about 12; R is hydrogen, alkyl or substituted alkyl with from 1 to about 6 carbon atoms, a polyethoxy group of the formula

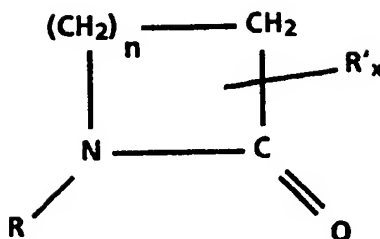


with m being a number of from 0 to about 9, or a polyimine group of the formula

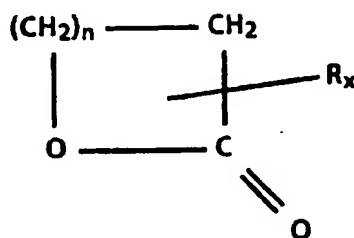


with k being a number of from 0 to about 9; R' represents one or more substituents that can replace hydrogen in any of the hydrocarbon portions of the molecule, and is an alkyl group, a halogen atom, a sulfate group, a nitro group, a sulfone group, an amide group, or an acetyl group, wherein x is a number of from 0 to 2n + 2;

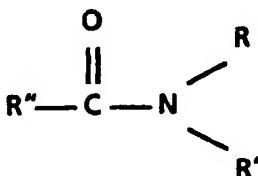
(2) cyclic esters of the formula



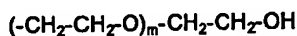
wherein n is a number of from 1 to about 12, R is a cyclohexyl group or a butyl group, and R' represents one or more substituents that can replace hydrogen in any of the hydrocarbon portions of the molecule, and is an alkyl group, a halogen atom, a sulfate group, a nitro group, a sulfone group, an amide group, or an acetyl group, wherein x is a number of from 0 to $2n + 2$; (3) cyclic esters of the formula



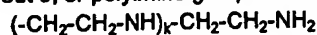
wherein n is a number of from 1 to about 12, R represents one or more substituents that can replace hydrogen in any of the hydrocarbon portions of the molecule, and is an alkyl group, a halogen atom, a sulfate group, a nitro group, a sulfone group, an amide group, or an acetyl group, wherein x is a number of from 0 to $2n + 2$; (4) amides of the formula



wherein R and R' are alkyl groups or substituted alkyl groups with from 2 to about 20 carbon atoms, polyethoxy groups of the formula



with m being a number of from 0 to about 9, or polyimine groups of the formula



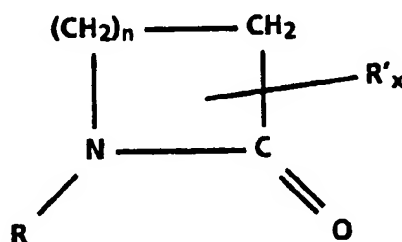
with k being a number of from 0 to about 9, wherein R and R' can be bonded to each other to form a ring, wherein R can also be hydrogen and wherein R'' is hydrogen or alkyl, with alkyl preferably having from 1 to about 20 carbon atoms; and mixtures thereof; and heating selected nozzles in the printing apparatus containing the ink, thereby causing droplets of the ink to be ejected in an imagewise pattern onto a substrate.

Inks employed in the printing processes of the present invention contain a colorant. Generally, any effective dye, such as one of the direct dyes or the acid dyes, can be selected as the colorant, provided that it is compatible with the other ink components and is soluble in the liquid vehicle. The dye is present in the ink composition in any effective amount, generally from about 1 to about 15 percent by weight, and preferably from about 2 to about 7 percent by weight (these values refer to the concentration of dye molecules in the ink), although the amount can be outside of this range.

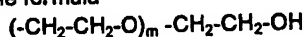
In addition, the colorant for the ink compositions of the present invention can be a pigment, or a mixture of one or more dyes and/or one or more pigments. The pigment can be black, cyan, magenta, yellow, red, blue, green, brown, mixtures thereof, and the like. Examples of suitable black pigments include various carbon blacks such as channel black, furnace black, lamp black, and the like. Colored pigments include red, green, blue, brown, magenta, cyan, and yellow particles, as well as mixtures thereof. Preferably, the pigment particle size

is as small as possible to enable a stable colloidal suspension of the particles in the liquid vehicle and to prevent clogging of the ink channels when the ink is used in a thermal ink jet printer. Preferred particle average diameters are generally from about 0.001 to about 0.1 micron, and more preferably from about 0.001 to about 0.01 microns, although the diameter can be outside of this range. The pigment is present in the ink composition in any effective amount, generally from about 1 to about 7 percent by weight and preferably from about 2 to about 5 percent by weight, although the amount can be outside of this range.

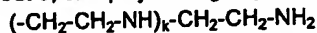
The ink compositions of the present invention contain a liquid vehicle which comprises a mixture of water and an organic component. One group of suitable organic components includes cyclic amides of the formula



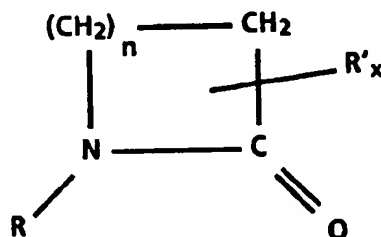
wherein n is a number of from 2 to about 12; R is hydrogen, alkyl or substituted alkyl with from 1 to about 6 carbon atoms, a polyethoxy group of the formula



with m being a number of from 0 to about 9, or a polyimine group of the formula

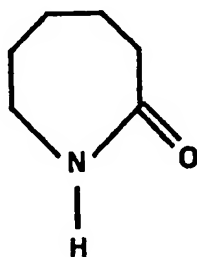


with k being a number of from 0 to about 9; R' represents one or more substituents that can replace hydrogen in any of the hydrocarbon portions of the molecule, and is an alkyl group (typically with from 1 to about 12 carbon atoms), a halogen atom, a sulfate group, a nitro group, a sulfone group, an amide group, or an acetyl group, wherein x is a number of from 0 to $2n + 2$; and a cyclic amide of the formula

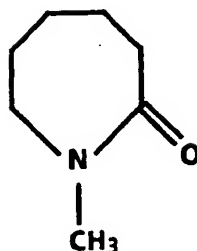


wherein n is a number of from 1 to about 12, R is a cyclohexyl group or a butyl group, and R' represents one or more substituents that can replace hydrogen in any of the hydrocarbon portions of the molecule, and is an alkyl group (typically with from 1 to about 12 carbon atoms), a halogen atom, a sulfate group, a nitro group, a sulfone group, an amide group, or an acetyl group, wherein x is a number of from 0 to $2n + 2$;

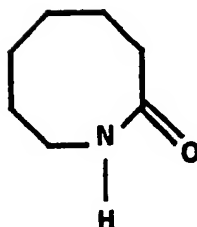
Specific examples of cyclic amides include of the formula ϵ -caprolactam,



methyl caprolactam, of the formula

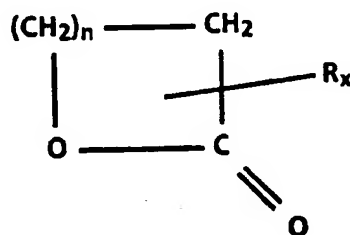


2-azacyclooctanone, of the formula



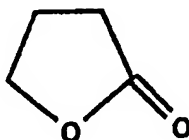
and the like. Compounds of these formulas are known, and are generally commercially available, such as cyclohexyl pyrrolidone, available from GAF, Wayne, NJ, ϵ -caprolactam, methyl caprolactam, 2-azacyclooctanone (8-membered ring), 2-azacyclononanone (9-membered ring), and 2-azacyclotridecanone (13-membered ring), available from Aldrich Chemical Company, Milwaukee, WI, and the like. Cyclic amides of these formulas are also available from BASF.

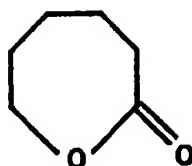
Another group of suitable organic components comprises cyclic esters of the formula



wherein n is a number from 1 to about 12, R represents one or more substituents that can replace hydrogen in any of the hydrocarbon portions of the molecule, and is an alkyl group (typically with from 1 to about 12 carbon atoms), a halogen atom, a sulfate group, a nitro group, a sulfone group, an amide group, or an acetyl group, wherein x is a number of from 0 to $2n+2$

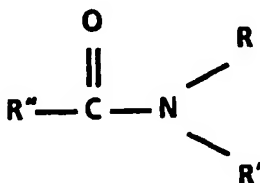
Specific examples of cyclic esters include butyrolactone, of the formula

 ϵ -caprolactone, of the formula

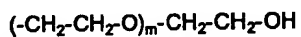


and the like. Compounds of these formulas are known, and are generally commercially available, such as butyrolactone and ϵ -caprolactone, available from Eastman Kodak Company, Rochester, NY or Aldrich Chemical Company, Milwaukee, WI.

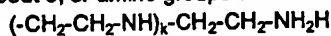
Another group of suitable organic components comprises amides of the formula



wherein R and R' are alkyl groups or substituted alkyl groups with from 2 to about 20 carbon atoms, ethoxy groups of the formula

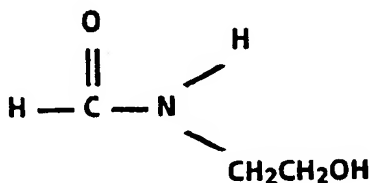


with m being a number of from 0 to about 9, or amino groups of the formula

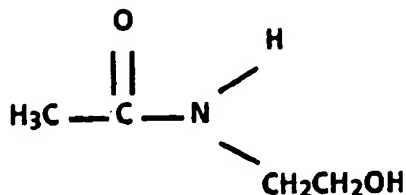


with k being a number of from 0 to about 9, wherein R and R' can be bonded to each other to form a ring, wherein R can also be hydrogen and wherein R'' is hydrogen or methyl. The amides suitable for the present invention can also have one or more substituents, such as alkyl groups (typically with from 1 to about 12 carbon atoms), halogen atoms, sulfate groups, nitro groups, sulfone groups, amide groups, acetyl groups or the like.

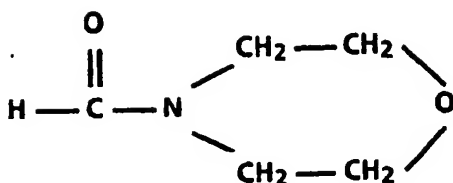
Specific examples of formamides include formylethanoamine, of the formula



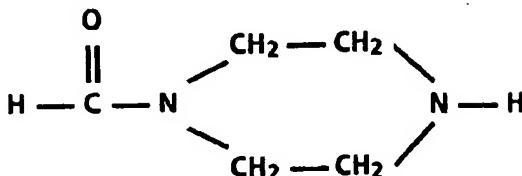
acetylethanolamine, of the formula



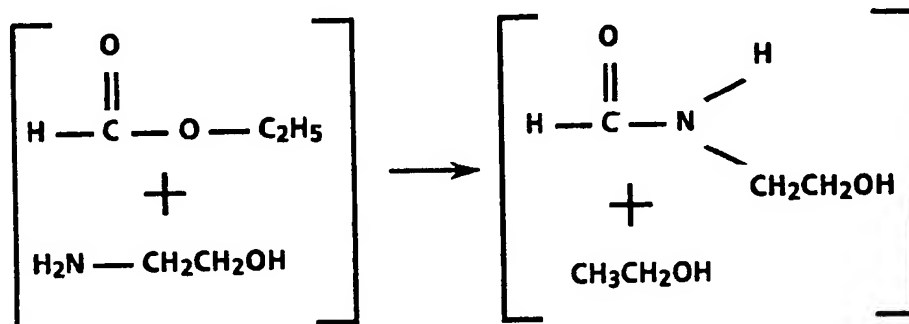
formylmorpholine, of the formula



formylpiperidine, of the formula



and the like. Compounds of these formulas are known, and are generally commercially available, such as acetylethanolamine, formylmorpholine, and formylpiperidine, available from Aldrich Chemical Company, Milwaukee, WI. In addition, compounds such as formylethanolamine can be prepared by mixing ethyl formate and monoethanolamine together in approximately a 1 to 1 molar ratio and heating the mixture to about 80°C to boil off the ethanol generated by the reaction, thus driving the reaction forward, as shown:



Similar compounds can be prepared from other alkyl formates, such as propyl formate, butyl formate, and the like, and other alkanol amines, such as propanolamine, butanolamine, and the like.

The materials selected for the organic component of the liquid vehicle are hygroscopic liquids or solids. Typically, many of the cyclic amides are solid at room temperature (typically from about 15 to about 35°C), such as the caprolactams, the azooctanones, and the like. The hygroscopic nature of these solids renders them readily soluble in water when the liquid vehicle of the ink is prepared.

The liquid vehicle of the inks employed for the present invention can contain water and the organic component in any effective amounts. Generally, the liquid vehicle comprises water in an amount of from about 50 to about 99.5 percent by weight, preferably from about 80 to about 99 percent by weight, and the organic component in an amount of from about 0.5 to about 50 percent by weight, preferably from about 1 to about 30 percent by weight, although the relative amounts of water and organic component can be outside of these ranges.

Other additives can also be present in the inks of the present invention. For example, when pigment particles are employed as the colorant, the colloidal stability of the pigment particles can be enhanced by the addition of one or more surfactants or wetting agents to the ink. Surfactants can also enhance the drying times of both pigmented and dyed inks. These additives may be of the cationic, anionic, or nonionic types. Suitable surfactants and wetting agents include Tamol® SN, Tamol® LG, those of the Triton® series available from Rohm and Haas Company, those of the Marasperse® series, those of the Igepal® series available from GAF Company, those of the Tergitol® series, and other commercially available surfactants. Other suitable surfactants include aliphatic sulfates, aromatic sulfonates, amine oxides, alkanol amides, and the like. These surfactants and wetting agents are present in effective amounts, generally from about 0.001 to about 5 percent by

weight, and preferably from about 0.01 to about 0.5 percent by weight, although the amount can be outside this range. The surfactants or wetting agents coat or adsorb onto the surfaces of the pigment particles to create electrically charged species, and also provide steric hindrance that separates the pigment particles, thereby enabling a stable colloidal dispersion.

5 Polymeric additives can also be added to the inks of the present invention to enhance the viscosity of the ink and the stability of pigment particles and to reduce the rate of agglomeration and precipitation of the particles. Water soluble polymers such as Gum Arabic, polyacrylate salts, polymethacrylate salts, polyvinyl alcohols, hydroxy propylcellulose, hydroxyethylcellulose, polyvinylpyrrolidinone, polyvinylether, starch, polysaccharides, and the like are particularly useful for stabilizing pigment particles in a water based liquid vehicle
10 such as water or a mixture of water and a water miscible organic liquid. Polymeric stabilizers may be present in the ink of the present invention in any effective amount, generally from 0 to about 10 percent by weight, and preferably from 0 to about 5 percent by weight, although the amount can be outside of this range.

Other optional additives to the inks prepared by the process of the present invention include biocides such as Dowicil 150, 200, and 75, benzoate salts, sorbate salts, and the like, present in an amount of from 0 to about
15 4 percent by weight, and preferably from about 0.01 to about 2.0 percent by weight, although the amount can be outside of this range, humectants such as ethylene glycol, diethyleneglycol, N-methylpyrrolidinone, propylene glycol, hydroxyethers, ethers, amides, sulfoxides, ketones, lactones, esters, alcohols, and the like, present in an amount of from 0 to about 50 percent by weight, and preferably from 0 to about 40 percent by weight, although the amount can be outside of this range, pH controlling agents such as acids or bases, phosphate
20 salts, carboxylates salts, sulfite salts, amine salts, and the like, present in an amount of from 0 to about 1 percent by weight and preferably from 0 to about 1 percent by weight, although the amount can be outside of this range, or the like.

In addition, the ink compositions of the present invention can contain one or more drying agents. Preferred drying agents are materials that exhibit moderate to strong hydrogen bonding. Examples of drying agents include sodium lauryl sulfate, such as Lomar D, available from E.I. Du Pont de Nemours & Company, Wilmington, DE, or Daxad 19K, available from W.R. Grace Company, Lexington, MA, sodium isopropyl naphthalene sulfonate, sulfones, such as dimethyl sulfone and the like, diamides, such as urea, 2-imidazolidone, and the like, amines, such as ethanolamine, piperazine, 2-pyrrolidone, dimethylurea, imidazole, pyrazole, 1,2,4-triazole, methyl isourea, diamino-2-propanol, diethanolamine, and the like, pseudoamines, such as 3-hydroxypyridine,
30 2-hydroxypyridine, and the like, acids, such as acetic acid and the like, inorganic bases, such as sodium hydroxide, potassium hydroxide, lithium hydroxide, calcium hydroxide, magnesium hydroxide, ammonium hydroxide, and the like, and any other suitable hydrogen bonding material, such as 2-hydroxyethyl pyridine, N-formylpiperazine, tetrazole, 2-methylimidazole, methylpyrazole, or the like. Particularly preferred drying agents are pyrazole and methylpyrazole, particularly 3-methyl pyrazole, as they are small, simple, thermally stable molecules that are not affected by pH over the range of 6 to 8. Additional examples of drying agents include N-coco alkyl-2,2'-iminobis ethanol N-oxide, available as Armox C/12 from Akzo Chemie, Chicago, IL, monoethanol amide, available as Ninol 1301 from Stepan Chemical Company, Northfield, IL, lauramide diethanol amine, available as Ninol 70-SL from Stepan Chemical Company, Northfield, IL, lauramide diethanol amine, available as Ninol 96-SL from Stepan Chemical Company, Northfield, IL, coco alkyldimethylamines oxide (30 percent solution in isopropanol), available as Armox DMC from Akzo Chemie, Chicago, IL, cocoamidopropylamine oxide (30 percent solution in isopropanol), available as Ammonyx CDO from Stepan Chemical Company, Northfield, IL, lauramide oxide (40 percent solution in isopropanol), available as Ammonyx DMCD-40 from Stepan Chemical Company, Northfield, IL, sodium dioctyl sulfosuccinate, available as Aerosol OT-100 from American Cyanamid Company, Wayne, NJ, isopropyl amine (branched) dodecyl benzene sulfonate (branched), available as Polystep A-11 from Stepan Chemical Company, Northfield, IL, amine alkyl benzene sulfonate (branched), available as Ninate 411 from Stepan Chemical Company, Northfield, IL, and the like. The drying agent can be present in any effective amount. Typical amounts for the drying agent are from about 0.001 to about 5 percent by weight, and preferably from about 0.01 to about 0.5 percent by weight, although the amount can be outside of this range.

50 Ink compositions of the present invention can be prepared by various methods, including simple mixing of the ingredients under ambient conditions and in the relative amounts desired in the ink. When the colorant is a pigment instead of a dye, one suitable ink preparation process entails mixing the selected pigment with water and, optionally, a wetting agent such as those of the Daxad® series of W.R. Grace (Daxad® 19, 19K, 11, 11 KIs, and the like, those of the Lomar® series, including Lomar® D, Lomar® W, and the like, those of the Igepal series, such as Igepal® Co 540, Co-560, and the like, those of the Tamol® series, such as Tamol SN and the like, those of the Triton® series, such as Triton 100 and the like, or organic liquids such as glycol derivatives, including ethylene glycol, diethylene glycol, polyethylene glycol, polypropylene glycol, and the like, with the wetting agent, if present, in the mixture in an amount of from about 0.1 to about 50 percent by weight to wet the
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pigment in the aqueous medium. Wetting enables the pigment to break apart to form a homogeneous aqueous dispersion. This mixture is then attrited, for example by placing it in a ball mill. The attrition can be performed in air or under an inert atmosphere. Attrition generally continues for a period of from about 0.25 to about 48 hours, which results in reduction of the pigment particle size to a desirable range of from about 0.0010 to about 20 microns in average particle diameter. Subsequent to attrition, the resulting dispersion of pigment particles can be subjected to filtration and/or centrifugation to remove undesirably large particles, and the dispersion can then be mixed with the organic component and any other desired ingredients to form an ink. Alternatively, the organic component and any other desired ingredients can be present in the dispersion of pigment particles prior to attrition, and subsequent to attrition and any desired filtration or centrifugation steps, the desired ink is obtained.

The present invention is also directed to a printing process which comprises applying an ink according to the present invention to a substrate in imagewise fashion. In a specific embodiment, the present invention includes a process which comprises incorporating into an ink jet printing apparatus an ink composition according to the present invention and causing droplets of the ink to be ejected in an imagewise pattern onto a substrate, thereby generating images on the substrate. The ink compositions of the present invention can be used to form images on a wide variety of substrates, including plain papers such as Xerox® 4024 papers, Xerox® 4200 papers, Xerox® 10 series paper, canary ruled paper, ruled notebook paper, bond paper such as Gilbert 25 percent cotton bond paper, Gilbert 100 percent cotton bond paper, and Strathmore bond paper, silica coated papers such as Sharp Company silica coated paper, JuJo paper, and the like, transparency materials such as Xerox® 3R3351, Tetrionix ink jet transparencies, Arkright ink jet transparencies, and the like, fabrics, textile products, plastics, polymeric films, inorganic substrates such as metals and wood, and the like.

Inks of the present invention exhibit characteristics that make them highly desirable for printing processes, particularly thermal ink jet printing processes. For example, the surface tension of inks of the present invention generally ranges from about 30 to about 65 dynes per centimeter, and preferably is as high as possible, preferably being from about 40 to about 65 dynes per centimeter and more preferably about 65 dynes per centimeter. Viscosity generally ranges from about 1.0 to about 5.0 centipoise and preferably ranges from 1 to 3 centipoise. Latency, which is the period during which the ink jet may be stopped while it contains the ink and subsequently restarted without clogging of the nozzle, is generally above 100 seconds, and frequently extends beyond 1000 seconds. Latency in general should be as high as possible, and at least 50 seconds, to enable restarting of the ink jet printer after extended idle periods. Prints made with the inks of the present invention generally exhibit image edge sharpness and reduced feathering on plain paper as well as treated or coated papers and transparencies. In addition, prints made from the inks of the present invention exhibit improved drying times, with the drying times on plain paper being as little as one second or less in some instances.

Specific embodiments of the invention will now be described in detail. These examples are intended to be illustrative, and the invention is not limited to the materials, conditions, or process parameters set forth in these embodiments. All parts and percentages are by weight unless otherwise indicated.

EXAMPLE I

Four ink compositions were prepared as follows. A concentrated aqueous dispersion of carbon black particles was prepared by adding to a Union Process attritor containing 1,500 grams of 1/8 inch diameter steel shots 176.5 grams of distilled water. To 50 additional grams of distilled water was added 13.5 grams of Lomar D (dispersing agent, a condensate of formaldehyde and sodium naphthalene sulfonate, obtained from E.I. Du Pont de Nemours & Company, Wilmington, DE). To the water in the attritor was then added 23 grams of the mixture of water and dispersing agent, and the attritor contents were mixed. Subsequently, 60 grams of Raven® 5250 carbon black (obtained from Columbian Specialty Chemicals Company, Atlanta, GA) was added to the attritor and the mixture was stirred until the carbon black was uniformly dispersed. Thereafter, the remaining 40.5 grams of the mixture of water and dispersing agent were added to the attritor while mixing was continued. After addition was complete, the attritor contents were attrited for 30 minutes, followed by removal of the dispersion by pipette to dryness. The inside of the attritor and the steel shots were then rinsed with 25 grams of distilled water and the water was added to the dispersion collected from the attritor. The rinsing process was repeated with another 25 grams of water, resulting a total recovery of 330 grams of dispersion from the attritor (94.39 percent yield, containing 17.14 percent by weight of carbon black solids). Samples of this carbon black dispersion were then mixed with water and various organic components to result in inks with the following compositions:

- A. 5 percent by weight of carbon black
- 1 percent by weight of Lomar D
- 15 percent by weight of methylcaprolactam (obtained from Aldrich Chemical Company, Milwaukee,

WI)

79 percent by weight of water

B. 5 percent by weight of carbon black

1 percent by weight of Lomar D

10 percent by weight of caprolactam (obtained from Aldrich Chemical Company, Milwaukee, WI)

84 percent by weight of water

C. 5 percent by weight of carbon black

1 percent by weight of Lomar D

15 percent by weight of 2-azacyclooctanone (obtained from Aldrich Chemical Company, Milwaukee,

WI)

79 percent by weight of water

D. 5 percent by weight of carbon black

1 percent by weight of Lomar D

15 percent by weight of caprolactone (obtained from Aldrich Chemical Company, Milwaukee, WI)

79 percent by weight of water

The inks were incorporated into a thermal ink jet test fixture equipped with a 48 jet printhead with a resolution of 288 spi and operated at 33.5 V with a pulse length of 3 microseconds to generate 100 drops per second. The inks were jetted under 15 percent relative humidity conditions and at 25°C, with air of the specified relative humidity and temperature being blown onto the surface of the printhead during jetting. Latency times were measured by jetting the inks at 100 drops per second, ceasing the jetting process for a given number of seconds, subsequently again applying voltage, and determining whether ink was jetted after the "latent" period. Latency times in seconds for inks A through D were as follows:

Ink	First Drop Latency (sec.)	Drop # 10 Latency (sec.)
A	75	> 1,000
B	75	> 1,000
C	50	> 1,000
D	50	200

The data for first drop latency were measured by allowing the printhead containing the ink to remain idle for the indicated time period, followed by jetting a single drop of ink; these times represent the amount of time that the printhead could remain idle followed by successful jetting of the first drop of ink from the printhead. The data for drop #10 latency were measured by allowing the printhead containing the ink to remain idle for the indicated time period, followed by jetting 10 drops of ink; these times represent the amount of time that the printhead could remain idle followed by successful jetting of the tenth drop of ink from the printhead. A typical maintenance procedure for printheads containing ink that have remained idle for a period of time is to jet 10 drops from each nozzle; when firing 10 drops from the nozzle fails to restore normal operation, a hard stop failure has occurred. As the data indicate, the inks of the present invention were able to remain in an idle printhead for periods of from 50 to 75 seconds, followed by successful jetting of the first drop fired from the nozzle. The inks of the present invention were also able to remain in an idle printhead for periods of from 200 to over 1,000 seconds, followed by successful jetting of the tenth drop fired from the nozzle. The high latency periods of these inks indicates that printheads containing these inks would require very little maintenance to restore normal operations after extended idle periods.

EXAMPLE II

An ink composition was prepared by mixing together by stirring a sample of the carbon black dispersion prepared in Example I with distilled water, an organic component, and a drying agent to result in an ink with the following composition:

E. 5 percent by weight of carbon black

1 percent by weight of Lomar D

25 percent by weight of 2-azacyclooctanone (obtained from Aldrich Chemical Company, Milwaukee,

WI)

2 percent by weight of 3-methyl pyrazole (drying agent, obtained from Aldrich Chemical Company, Milwaukee, WI)

67 percent by weight of water

5 Ink E as well as inks A and D from Example I were subjected to an extended latency test by incorporating each ink into the printing test fixture of Example I and determining the number of drop ejection attempts required to restore normal jetting functioning after extended idle periods. The number of drops required to restore normal functioning under the indicated conditions were as follows:

10	Ink	Quiescent Condition	Drop #
	A	1 hour at 20% relative humidity	5
	A	19 hours at 20% relative humidity	16
15	D	10 minutes at 20% relative humidity	190
	E	19 hours at 18% relative humidity	350

20 As the data indicate, inks of the present invention enabled normal jetting function to be restored even after idle periods of 19 hours. In contrast, typical ink compositions containing carbon black pigment particles result in complete clogging of the jetting nozzles after as little as 1 hour, after which normal jetting functions cannot be restored.

25 EXAMPLE III

Ink compositions were prepared as follows. Samples of the carbon black dispersion prepared in Example I were mixed with water and organic components (solids in this instance) in varying amounts to produce ink compositions containing 6 percent by weight of carbon black and the amount of organic component shown in the table below, with the balance of the composition being water. A control sample was also prepared containing 6 percent by weight of carbon black and 94 percent by weight of water. Each ink was hand coated onto the wire (porous) and felt (sized) sides of Xerox® '10' Series Smooth paper using a #8 Meyer rod for solid areas and a simple ruling pen for lines. The dry time for each ink was determined by noting the time required for the coating to show negligible smearing when abraded with a Q-tip®. Drying times for inks containing the indicated organic components in the indicated amounts were as follows:

35 As the data indicate, the organic components, which as shown in Examples I and II improve

40	Organic Component and Concentration (percent by weight)	Drying Time - Felt Side (seconds)	Drying Time - Wire Side (seconds)
	Control (no organic component)	22	16
45	methyl caprolactam - 5%	19	15
	methyl caprolactam - 10%	22	13
	methyl caprolactam - 20%	29	12
50	2-azacyclooctanone - 10%	24	18
	2-azacyclooctanone - 15%	26	17
	2-azacyclooctanone - 20%	28	16
55	2-azacyclooctanone - 25%	26	15

latency times, in many instances also improved drying times. The drying times also frequently appeared to im-

prove (decrease) with increasing organic component concentration, which is in contrast to other commonly used ink jet organic components such as ethylene glycol, for which drying is retarded as concentration increases. In addition, the ratio of dry times on the felt and wire sides at a given concentration is also of interest, since the closer this ratio is to unity the less variability in dry time would be expected over a range of sized and porous papers. In this instance, 10 percent of 2-azacyclooctanone exhibited outstanding performance with a ratio of 1:1.3.

EXAMPLE IV

Ink compositions were prepared as follows. A concentrated aqueous dispersion of carbon black particles was prepared by adding to a Union Process attritor containing 1,500 grams of 1/8 inch diameter steel shots 175 grams of distilled water. To 50 additional grams of distilled water was added 15 grams of Daxad 19k (dispersing agent, a condensate of formaldehyde and sodium naphthalene sulfonate, obtained from W.R. Grace & Company). To the water in the attritor was then added 23 grams of the mixture of water and dispersing agent, and the attritor contents were mixed. Subsequently, 60 grams of Raven® 5250 carbon black (obtained from Columbian Specialty Chemicals Company, Atlanta, GA) was added to the attritor and the mixture was stirred until the carbon black was uniformly dispersed. Thereafter, the remaining 42 grams of the mixture of water and dispersing agent were added to the attritor while mixing was continued. After addition was complete, the attritor contents were attrited for 30 minutes, followed by removal of the dispersion by pipette to dryness. The inside of the attritor and the steel shots were then rinsed with 25 grams of distilled water and the water was added to the dispersion collected from the attritor. The rinsing process was repeated with another 25 grams of water, which was also added to the dispersion collected from the attritor. Samples of the carbon black dispersion thus produced were then mixed with water and various organic components to result in inks with the following compositions:

- 25 F. 6 percent by weight of carbon black
 - 1.5 percent by weight of Daxad 19k
 - 10 percent by weight of methyl caprolactam
 - 5 percent by weight of 3-methyl pyrazole
 - 77.5 percent by weight of water
- 30 G. 6 percent by weight of carbon black
 - 1.5 percent by weight of Daxad 19k
 - 10 percent by weight of methyl caprolactam
 - 8 percent by weight of 3-methyl pyrazole
 - 74.5 percent by weight of water
- 35 H. 6 percent by weight of carbon black
 - 1.5 percent by weight of Daxad 19k
 - 10 percent by weight of methyl caprolactam
 - 5 percent by weight of pyrazole
 - 77.5 percent by weight of water
- 40 I. 6 percent by weight of carbon black
 - 1.5 percent by weight of Daxad 19k
 - 10 percent by weight of methyl caprolactam
 - 7.5 percent by weight of cyclohexyl pyrrolidone
 - 75 percent by weight of water
- 45 J. 6 percent by weight of carbon black
 - 1.5 percent by weight of Daxad 19k
 - 20 percent by weight of methyl caprolactam
 - 10 percent by weight of pyrazole
 - 62.5 percent by weight of water
- 50 K. 6 percent by weight of carbon black
 - 1.5 percent by weight of Daxad 19k
 - 20 percent by weight of methyl caprolactam
 - 5 percent by weight of pyrazole
 - 67.5 percent by weight of water
- 55 L. 6 percent by weight of carbon black
 - 1.5 percent by weight of Daxad 19k
 - 20 percent by weight of methyl caprolactam
 - 5 percent by weight of 3-methyl pyrazole

- 67.5 percent by weight of water
 M. 6 percent by weight of carbon black
 1.5 percent by weight of Daxad 19k
 20 percent by weight of methyl caprolactam
 4 percent by weight of cyclohexyl-2-pyrrolidone
 68.5 percent by weight of water
- N. 6 percent by weight of carbon black
 1.5 percent by weight of Daxad 19k
 20 percent by weight of methyl caprolactam
 2 percent by weight of (AR) Ammonyx CDO (surfactant, obtained from Stepan Chemical Company, Northfield, IL)
 70.5 percent by weight of water
- O. 6 percent by weight of carbon black
 1.5 percent by weight of Daxad 19k
 20 percent by weight of methyl caprolactam
 0.3 percent by weight of sodium lauryl sulfate
 72.2 percent by weight of water
- P. 6 percent by weight of carbon black
 1.5 percent by weight of Daxad 19k
 10 percent by weight of caprolactam
 10 percent by weight of pyrazole
 72.5 percent by weight of water
- Q. 6 percent by weight of carbon black
 1.5 percent by weight of Daxad 19k
 10 percent by weight of 2-azacyclooctanone
 5 percent by weight of pyrazole
 77.5 percent by weight of water
- R. 6 percent by weight of carbon black
 1.5 percent by weight of Daxad 19k
 10 percent by weight of 2-azacyclooctanone
 3 percent by weight of 3-methyl pyrazole
 79.5 percent by weight of water
- S. 6 percent by weight of carbon black
 1.5 percent by weight of Daxad 19k
 10 percent by weight of butyrolactone
 5 percent by weight of pyrazole
 77.5 percent by weight of water
- T. 6 percent by weight of carbon black
 1.5 percent by weight of Daxad 19k
 10 percent by weight of caprolactone
 5 percent by weight of pyrazole
 77.5 percent by weight of water
- U. 6 percent by weight of carbon black
 1.5 percent by weight of Daxad 19k
 5 percent by weight of formyl ethanol amine
 5 percent by weight of acetyl ethanol amine
 5 percent by weight of pyrazole
 77.5 percent by weight of water
- V. 6 percent by weight of carbon black
 1.5 percent by weight of Daxad 19k
 15 percent by weight of formyl ethanol amine
 5 percent by weight of pyrazole
 72.5 percent by weight of water
- W. 6 percent by weight of carbon black
 1.5 percent by weight of Daxad 19k
 15 percent by weight of formyl morpholine
 10 percent by weight of pyrazole
 67.5 percent by weight of water

These ink compositions were incorporated into a thermal ink jet test fixture equipped with a 48 jet printhead with a resolution of 288 spi and operated at 33.5 V with a pulse length of 3 microseconds to generate 100 drops per second. The inks were jetted at 25°C, with air of the specified relative humidity and temperature being blown onto the surface of the printhead during jetting. Latency times were measured by jetting the inks at 100 drops per second, ceasing the jetting process for a given number of seconds, subsequently again applying voltage, and determining whether ink was jetted after the "latent" period. The dry time for each ink was determined by noting the time required for the coating to show negligible smearing when abraded with a Q-tip®. Latency times and dry times in seconds for inks F through W were as follows:

Ink	% RH	Latency (sec.)		FMAX (KHz)	Solid Area Dry Time		Text Area Dry Time	
		#1 Drop	#10 Drop		Wire	Felt	Wire	Felt
F		40	500	3.1	7	16	<3	8
G	24	50	<200	3.0	6	9	<3	4
H		100	>500	4.2	9	17	6	8
I	57	75	>1,000	3.6	13	17	6	8
J	39	40	500	2.4	6	15	<3	8
K	42	>1,000	>1,000	3.0	9	21	<4	7
L	23	40	500	2.4	9	17	<4	6
M	39	75	>1,000	2.4	13	20	<4	13
N	23	75	>100	2.4	9	23	6	11
O	24	>1,000	>1,000	3.0	<6	18	<3	9
P	62	--	--	3.0	11	22	6	9
Q	35	75	<200	3.8	15	19	6	9
R	--	--	--	--	10	17	4	7
S	--	20	30	5.5	16	17	6	6
T	--	30	50	--	14	18	8	9
U	--	30	75	--	21	25	9	11
V	--	20	50	--	18	26	8	10
W	--	20	50	4.8	19	29	8	17

(-- indicates that the value was not measured)

As the data indicate, inks of the present invention enable excellent drying times, latency times, or both. The improved drying times enable the use of these inks in printing apparatuses without smearing of the images formed as the printed sheets pass through the machine and are stacked in an output tray. The improved latency times enable restarting of the printer and restoration of normal jetting operations after periods of idleness.

EXAMPLE V

Ink compositions were prepared by mixing portions of a carbon black dispersion prepared as described in Example I with water, methylcaprolactam, and a drying agent to result in inks of the following compositions:

- X. 5 percent by weight of carbon black
20 percent by weight of methylcaprolactam (obtained from Aldrich Chemical Company, Milwaukee, WI)
2 percent by weight of pyrazole (obtained from Aldrich Chemical Company, Milwaukee, WI)
73 percent by weight of water
- 5 Y. 5 percent by weight of carbon black
20 percent by weight of methylcaprolactam (obtained from Aldrich Chemical Company, Milwaukee, WI)
5 percent by weight of pyrazole (obtained from Aldrich Chemical Company, Milwaukee, WI)
70 percent by weight of water
- 10 Z. 5 percent by weight of carbon black
20 percent by weight of methylcaprolactam (obtained from Aldrich Chemical Company, Milwaukee, WI)
10 percent by weight of pyrazole (obtained from Aldrich Chemical Company, Milwaukee, WI)
65 percent by weight of water
- 15 AA. 5 percent by weight of carbon black
20 percent by weight of methylcaprolactam (obtained from Aldrich Chemical Company, Milwaukee, WI)
2 percent by weight of n-cyclohexyl pyrrolidone (obtained from Aldrich Chemical Company, Milwaukee, WI)
73 percent by weight of water
- 20 BB. 5 percent by weight of carbon black
20 percent by weight of methylcaprolactam (obtained from Aldrich Chemical Company, Milwaukee, WI)
5 percent by weight of n-cyclohexyl pyrrolidone (obtained from Aldrich Chemical Company, Milwaukee, WI)
70 percent by weight of water
- 25 CC. 5 percent by weight of carbon black
20 percent by weight of methylcaprolactam (obtained from Aldrich Chemical Company, Milwaukee, WI)
7 percent by weight of n-cyclohexyl pyrrolidone (obtained from Aldrich Chemical Company, Milwaukee, WI)
68 percent by weight of water
- 30 DD. 5 percent by weight of carbon black
20 percent by weight of methylcaprolactam (obtained from Aldrich Chemical Company, Milwaukee, WI)
10 percent by weight of n-cyclohexyl pyrrolidone (obtained from Aldrich Chemical Company, Milwaukee, WI)
65 percent by weight of water
- 35 EE. 5 percent by weight of carbon black
20 percent by weight of methylcaprolactam (obtained from Aldrich Chemical Company, Milwaukee, WI)
2 percent by weight of cocoamidopropylamine oxide (Ammonyx CDO, obtained from Stepan Chemical Company, Chicago, IL)
73 percent by weight of water
- 40 FF. 5 percent by weight of carbon black
20 percent by weight of methylcaprolactam (obtained from Aldrich Chemical Company, Milwaukee, WI)
5 percent by weight of cocoamidopropylamine oxide (Ammonyx CDO, obtained from Stepan Chemical Company, Chicago, IL)
70 percent by weight of water
- 50 GG. 5 percent by weight of carbon black
20 percent by weight of methylcaprolactam (obtained from Aldrich Chemical Company, Milwaukee, WI)
10 percent by weight of cocoamidopropylamine oxide (Ammonyx CDO, obtained from Stepan Chemical Company, Chicago, IL)
65 percent by weight of water
- 55 HH. 5 percent by weight of carbon black
20 percent by weight of methylcaprolactam (obtained from Aldrich Chemical Company, Milwaukee,

- WI)
0.5 percent by weight of sodium lauryl sulfate (obtained from Fisher Scientific Company, Fair Lawn,
NJ)
74.5 percent by weight of water
5 II. 5 percent by weight of carbon black
20 percent by weight of methylcaprolactam (obtained from Aldrich Chemical Company, Milwaukee,
WI)
1 percent by weight of sodium lauryl sulfate (obtained from Fisher Scientific Company, Fair Lawn,
NJ)
10 74 percent by weight of water
JJ. 5 percent y weight of carbon black
20 percent by weight of methylcaprolactam (obtained from Aldrich Chemical Company, Milwaukee,
WI)
5 percent by weight of sodium lauryl sulfate (obtained from Fisher Scientific Company, Fair Lawn,
15 NJ)
70 percent by weight of water

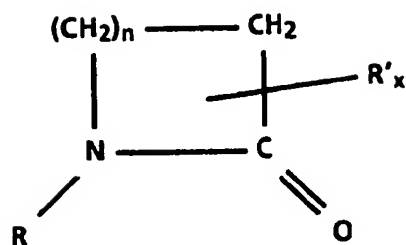
Each ink was hand coated onto the felt (sized) side of Xerox® '10' Series Smooth paper using a #8 Meyer rod for solid areas. The dry time for each ink was determined by noting the time required for the coating to show negligible smearing when abraded with a Q-tip®. Drying times for Inks X through JJ were as follows:

Ink	Drying Agent	Dry Time (sec.)
X	2% by weight pyrazole	19
Y	5% by weight pyrazole	16
Z	10% by weight pyrazole	12
AA	2% by weight n-cyclohexyl pyrrolidone	13
BB	5% by weight n-cyclohexyl pyrrolidone	10
CC	7% by weight n-cyclohexyl pyrrolidone	6
DD	10% by weight n-cyclohexyl pyrrolidone	2
EE	2% by weight Ammonyx COO	12
FF	5% by weight Ammonyx COO	2
GG	10% by weight Ammonyx COO	0.5
HH	0.5% by weight sodium lauryl sulfate	5
II	1% by weight sodium lauryl sulfate	2
JJ	5% by weight sodium lauryl sulfate	0.5

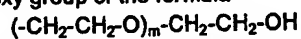
These data illustrate the additional improvements in drying times that can be obtained with inks of the present invention that contain added drying agents. The use of additional drying agents enables further control and "fine tuning" of the drying time for the ink composition.

Claims

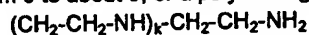
1. A thermal ink jet printing process which comprises incorporating into a thermal ink jet printing apparatus an ink composition comprising a colorant and a liquid vehicle which comprises a mixture of water and an organic component selected from the group consisting of: (1) cyclic amides of the formula



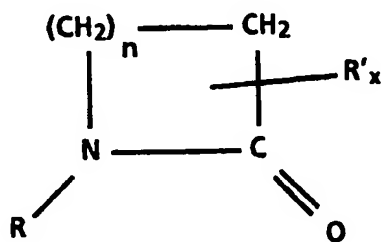
wherein n is a number of from 2 to about 12; R is hydrogen, alkyl or substituted alkyl with from 1 to about 6 carbon atoms, a polyethoxy group of the formula



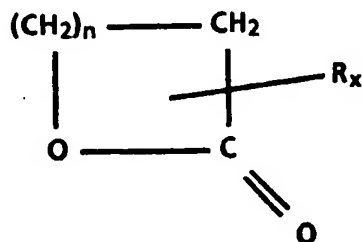
with m being a number of from 0 to about 9, or a polyimine group of the formula



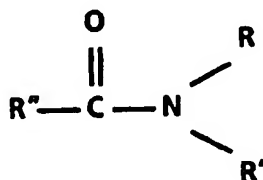
with k being a number of from 0 to about 9; R' represents one or more substituents that can replace hydrogen in any of the hydrocarbon portions of the molecule, and is an alkyl group, a halogen atom, a sulfate group, a nitro group, a sulfone group, an amide group, or an acetyl group, wherein x is a number of from 0 to $2n + n$; (2) a cyclic amide of the formula



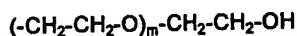
wherein n is a number of from 1 to about 12, R is a cyclohexyl group or a butyl group, and R' represents one or more substituents that can replace hydrogen in any of the hydrocarbon portions of the molecule, and is an alkyl group, a halogen atom, a sulfate group, a nitro group, a sulfone group, an amide group, or an acetyl group, wherein x is a number of from 0 to $2n + 2$; (3) cyclic esters of the formula



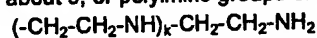
wherein n is a number of from 1 to about 12, R represents one or more substituents that can replace hydrogen in any of the hydrocarbon portions of the molecule, and is an alkyl group, a halogen atom, a sulfate group, a nitro group, a sulfone group, an amide group, or an acetyl group, wherein x is a number of from 0 to $2n + 2$; (4) amides of the formula



wherein R and R' are alkyl groups or substituted alkyl groups with from 2 to about 20 carbon atoms, polyethoxy groups of the formula

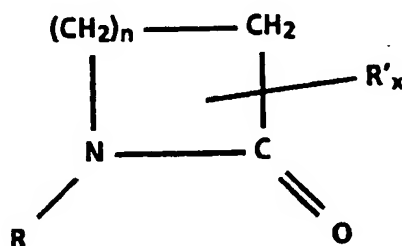


with m being a number of from 0 to about 9, or polyimine groups of the formula

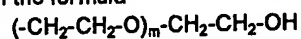


with k being a number of from 0 to about 9, wherein R and R' can be bonded to each other to form a ring, wherein R can also be hydrogen and wherein R' is hydrogen or alkyl; and mixtures thereof; and heating selected nozzles in the printing apparatus containing the ink, thereby causing droplets of the ink to be ejected in an imagewise pattern onto a substrate.

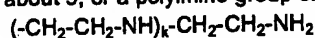
2. A thermal ink jet printing process according to claim 1 wherein the liquid vehicle comprises water in an amount of from about 50 to about 99.5 percent by weight and the organic component in an amount of from about 0.5 to about 50 percent by weight.
3. A thermal ink jet printing process according to claim 1 or claim 2 wherein the ink composition contains a surfactant in an amount of from about 0.001 to about 5 percent by weight.
4. A thermal ink jet printing process according to any one of claims 1 to 4 wherein the ink composition contains a drying agent in an amount of from about 0.001 to about 5 percent by weight.
5. A thermal ink jet printing process according to claim 4 wherein the drying agent is selected from: sodium lauryl sulfate, sodium isopropyl naphthalene sulfonate, sulfones, diamides, amines, pseudoamines, acids, inorganic bases, such as sodium hydroxide, ammonium hydroxide, dimethyl sulfone, urea, 2-imidazolidone, ethanolamine, piperazine, 2-pyrrolidone, dimethylurea, imidazole, pyrazole, 1,2,4-triazole, methyl isourea, diamino-2-propanol, diethanolamine, pseudoamines, such as 3-hydroxypyridine, 2-hydroxypyridine, 3-hydroxypyridine, 2-hydroxypyridine, acetic acid, sodium hydroxide, potassium hydroxide, lithium hydroxide, calcium hydroxide, magnesium hydroxide, ammonium hydroxide, 2-hydroxyethyl pyridine, N-formylpiperazine, tetrazole, 2-methylimidazole, methylpyrazole, N-coco alkyl-2,2'-iminobis ethanol N-oxide, monoethanol amide, lauramide diethanol amine, coco alkyl dimethylamines oxide, cocoamidopropylamine oxide, lauramide oxide, sodium dioctyl sulfosuccinate, isopropyl amine dodecyl benzene sulfonate, amine alkyl benzene sulfonate, and mixtures thereof.
6. A thermal ink jet printing process according to any of claims 1 to 5 wherein the organic component is a solid at temperatures of up to about 35°C.
7. An ink composition comprising a colorant and a liquid vehicle which comprises a mixture of water and an organic component comprising a cyclic amide of the formula



wherein n is a number from 2 to about 12; R is hydrogen, alkyl or substituted alkyl with from 1 to about 6 carbon atoms, a polyethoxy group of the formula



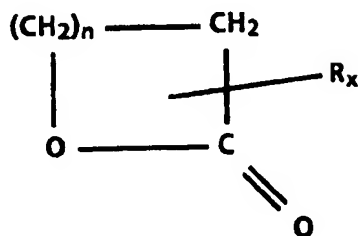
with m being a number of from 0 to about 9, or a polyimine group of the formula



with k being a number of from 0 to about 9; R' represents one or more substituents that can replace hydrogen in any of the hydrocarbon portions of the molecule, and is an alkyl group, a halogen atom, a sulfate group, a nitro group, a sulfone group, an amide group, or an acetyl group, wherein x is a number of from 0 to 2n + 2.

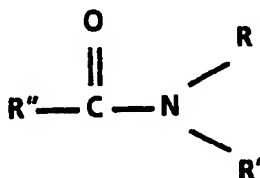
8. An ink composition comprising a colorant and a liquid vehicle which comprises a mixture of water and an

organic component comprising a cyclic ester of the formula

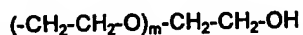


wherein n is a number of from 1 to about 12, R represents one or more substituents that can replace hydrogen in any of the hydrocarbon portions of the molecule, and is an alkyl group, a halogen atom, a sulfate group, a nitro group, a sulfone group, an amide group, or an acetyl group, wherein x is a number of from 0 to $2n + 2$.

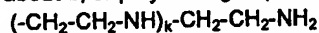
9. An ink composition comprising a colorant and a liquid vehicle which comprises a mixture of water and an organic component comprising an amide of the formula



wherein R and R' are alkyl groups or substituted alkyl groups with from 2 to about 20 carbon atoms, polyethoxy groups of the formula

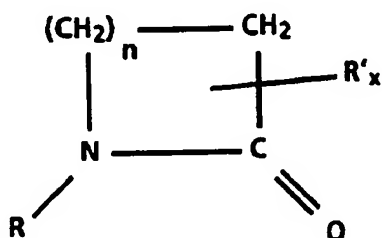


with m being a number of from 0 to about 9, or polyimine groups of the formula



with k being a number of from 0 to about 9, wherein R and R' can be bonded to each other to form a ring, wherein R can also be hydrogen and wherein R'' is hydrogen or alkyl.

10. An ink composition comprising a colorant and a liquid vehicle which comprises a mixture of water and an organic component comprising a cyclic amide of the formula



wherein n is a number of from 1 to about 12, R is a cyclohexyl group, and R' represents one or more substituents that can replace hydrogen in any of the hydrocarbon portions of the molecule, and is an alkyl group, a halogen atom, a sulfate group, a nitro group, a sulfone group, an amide group, or an acetyl group, wherein x is a number of from 0 to $2n + 2$.



European Patent
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EUROPEAN SEARCH REPORT

Application Number

EP 92 30 6950

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.5)
X	PATENT ABSTRACTS OF JAPAN vol. 13, no. 134 (C-581)(3482) 4 April 1989 & JP-A-63 297 477 (FUJI PHOTO FILM CO LTD) 5 December 1988 * abstract *	1,9	C09D11/00
X	DATABASE WPIL Week 8551, Derwent Publications Ltd., London, GB; AN 85321684 & JP-A-60 226 575 (SUMITOMO CHEM IND KK) 11 November 1985 * abstract *	1,8	
			TECHNICAL FIELDS SEARCHED (Int. Cl.5)
			C09D
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 27 NOVEMBER 1992	Examiner CATURLA VICENTE V.
<p>CATEGORY OF CITED DOCUMENTS</p> <p>X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document</p> <p>T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons</p> <p>..... A : member of the same patent family, corresponding document</p>			

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